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Measurement of the elevated temperature stress rupture characteristics of carbon fibres

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An apparatus for measurement of the stress rupture characteristics of six, individually mounted, carbon fibres at temperatures up to 650°C in vacuum is described. Some preliminary results on aluminium coated carbon fibres are reported.

1. Introduction

The carbon fibre reinforced aluminium composite system is potentially applicable at temperatures above 100°C and, as such, would have an advantage over reinforced polymer systems. However, as aluminium and carbon react to form a stable aluminium carbide, the maximum operating temperature is limited by any degradation in the ultimate tensile strength of the carbon fibres produced by the carbide reaction. This effect may be studied directly in a composite of the simplest form, namely an individual aluminium coated carbon fibre.

Two possible test methods for assessing the effect of heat-treatment on the mechanical properties of an individual fibre are:

(1) heat-treatment of the fibre, followed by a measurement of the room temperature ultimate tensile strength, as described previously for nickel coated carbon fibres [1];

(2) application of a constant load to the fibre at the annealing temperature, with measurement of the time to rupture (and any creep strain).

The results obtained from the first method are discussed elsewhere [2], while the second method required the development of novel apparatus. Two major requirements of such apparatus are an inert atmosphere to prevent oxidation of the carbon fibres and, in view of the strength distribution of brittle fibres, the ability to test several samples simultaneously. In this paper, the design of an apparatus for measuring the stress rupture and creep characteristics of six, individually mounted carbon fibres, in a vacuum of $< 10^{-5}$ Torr, at temperatures up to 650°C is reported, together with some preliminary results on the time to rupture of aluminium coated carbon fibres as a function of applied stress.

2. Description of apparatus

2.1. Load application and time to rupture and strain measurement

Fig. 1a shows the general arrangement of the load application and strain measuring system for six fibre specimens (only one of the six pairs of pull rods are connected in this figure). Each fibre, mounted on Nimonic adaptors, was stressed by the application of a dead weight to one end of a stainless steel beam, which was pivoted on a horizontal pin midway between the weight and a specimen pull rod (Fig. 1b). This pull rod was connected to the beam by an adjustable "point" contact and hung vertically over a similar rod mounted in a plate above the diffusion pump entry of the baseplate. The lower rod was equipped with a sliding sheath which maintained the specimen in a vertical position. Six of these systems were supported by their pivot mounts, at 60° separation, on an annulus mounted on three rods fixed to the chamber baseplate, such that the specimen pull rods hung on the circumference of a circle around the centre of the chamber. A load applicator disc was positioned above the inner ends of the beams with a rotatable cam above each beam, and was connected to a slow speed (3 Hz) electric motor. This was mounted on an annulus above the pivot support annulus connected to the three vertical rods. This arrangement provided adjustment for fixing a specimen

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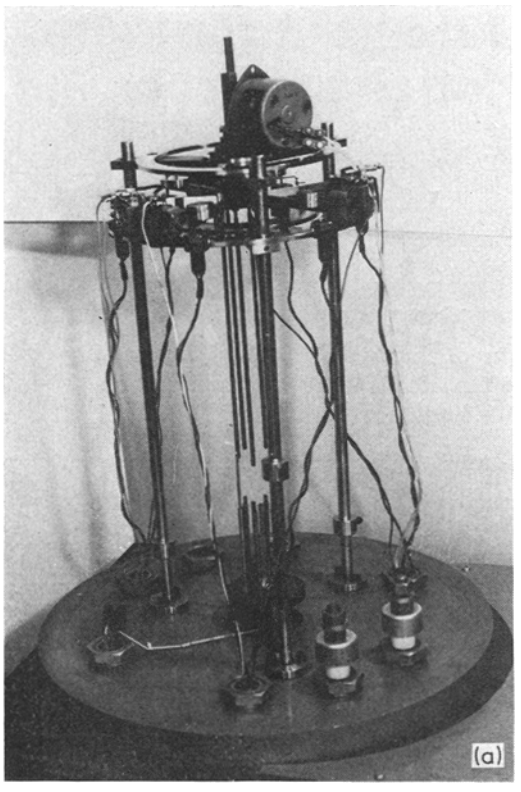

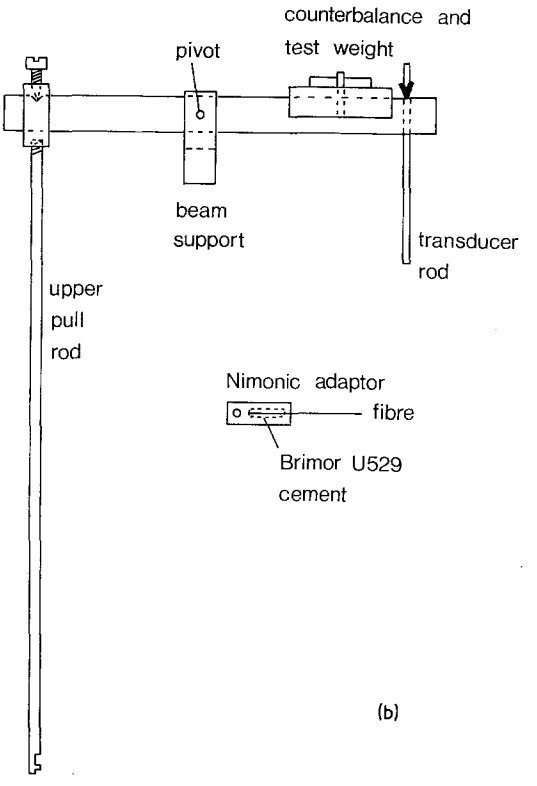

Figure 1 (a) General arrangement of the loading arrangement (with furnace, bell jar and implosion shield removed). (b) Individual fibre mounting system and loading beam (not to scale).

gauge length, held the load off the fibres until required, and applied the load at a strain rate slow enough not to fracture the specimens. Fibre specimens were mounted with Brimor U529 cement on Nimonic 75 adapators, which fitted on pins in the specimen pull rods and the lower rods.

At the outer end of each beam was a displacement transducer rod, supported by a "knife" edge, which penetrated into the Plessey transducer body mounted in an annulus of greater diameter, but on the same level as the pivot support annulus. The transducer rod mounting was adjustable to ensure that for each test temperature the penetration into the body would provide an "on-scale" reading on the C-52 meter. Mounted on the same annulus as the transducer bodies were six rotary microswitches, the levers of which passed beneath the beams and which were actuated when the specimens fractured. Each switch automatically switched off a Smiths 24 V d.c. 10 000 h clock to record the time-to-rupture of each specimen.

2.2. Furnace system

A furnace system was designed to surround the six fibre specimens. The furnace system (Fig. 2) was supported by its casing on the three support rods and could be raised such that the specimen pull rods passed completely through its length. The furnace core consisted of a solid 5 cm long

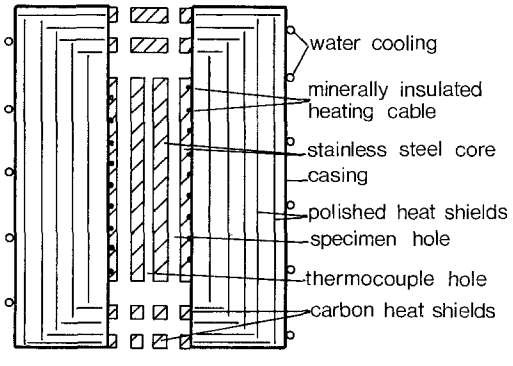

Figure 2 Schematic arrangement of the furnace system.

stainless steel rod, through which six holes had been drilled corresponding to the specimen pull rod positions, and a seventh hole bored centrally to accept a Pt 13% Rh thermocouple in an alumina sheath. A helical groove, machined in the edge of the core, accommodated 1 m of HSQ 10K minerally insulated heating wire. The core was contained in a thin walled stainless steel casing, with carbon heat shields above and below drilled to accept the specimen pull rods. This assembly was positioned centrally in the furnace casing and was surrounded by a system of polished molybdenum heat shields. Further cooling of the casing was provided by copper water pipes wound round the casing. The furnace temperature was controlled by a Eurotherm LP96 controller and recorded by a Chessel 301 potentiometric recorder both employing the same thermocouple.

2.3. Vacuum system

The load application and furnace system were covered by a glass bell jar, with a rubber seal to the nickel plated base plate (Fig. 1a) and the chamber evacuated. A vacuum of $< 10^{-5}$ Torr was obtained by a conventional diffusion pump system. An Edwards ES100 rotary pump was mounted beneath the equipment and connected to the vacuum piping system by a flexible connector to minimize vibration. The vacuum piping was then split, one part going via a valve to the main vacuum chamber and the other via another valve to the backing connection of an AE1 033C diffusion pump. This was mounted beneath an AE1 CT3 liquid nitrogen cold trap and an HB3 baffle valve which were positioned beneath a 5 cm diameter hole bored centrally through the baseplate. Protection from implosion was provided by an expanded aluminium guard. The backing pressure was monitored by an Edwards Pirani gauge mounted in the vacuum piping and the chamber pressure by an Edwards Penning gauge mounted through the system baseplate.

3. Experimental procedure

Carbon fibres ($\sim$ 8 μm in diameter) were coated by vapour deposition with 180 nm of aluminium [3]. From each fibre two sections were tested individually in an Instron testing machine and their mean ultimate tensile strength (unless one was badly flawed) used as a standard for the experiment. The third section of fibre was mounted on the Nimonic 75 adaptors by applying a drop of Brimor U529 cement to each adaptor while positioned in a gauge length (2.5 cm) maintaining jig. The cement was allowed to harden for 1 h under an electric light, after which it was baked successively for 1 h at 100, 200 and 250° C.

The furnace was then raised and the load applicator lowered to produce the correct gauge length, (individual adjustment was made using the specimen pull rod contact or the load applicator cam) and the microswitches reset. A mounted fibre was inserted on to the pins of the specimen pull rods. When all six fibres had been inserted, the required weights were applied to the beams. The furnace was then lowered into its operational position and the bell jar replaced.

The system was evacuated to $< 10^{-5}$ Torr and the load applied to the fibres by raising the load applicator disc. The furnace temperature was then raised slowly to 400° C to allow fibre outgassing and then rapidly to the required operating temperature, when the clocks were switched on. Periodic readings were taken from each of the transducers using a six-way switching unit between the transducers and the meter.

4. Experimental results

The performance of the stress rupture apparatus was evaluated by a series of tests on both uncoated and aluminium coated carbon fibres. In these tests, the times to rupture for uncoated and aluminium coated (180 nm thick) Courtaulds HT-S (high tensile strength-surface treated) fibres were measured at temperatures up to 650° C (at $\leqslant 10^{-5}$ Torr) for various levels of applied stress. The uncoated fibres did not rupture in times up to 500 h and at stresses up to 90% of the fibres' room temperature ultimate tensile strength when tested at 650° C. In contrast, as shown in Fig. 3, the time to rupture for the aluminium coated carbon fibres tested at 600 and 625° C decreased with an increase in the applied stress (expressed as a percentage of the room temperature ultimate tensile strength). This effect is attributed to the time dependence of aluminium carbide formation and discussed elsewhere [3].

5. Conclusions

The apparatus constructed allows simultaneous measurement of the stress rupture characteristics of six individually mounted, uncoated or aluminium coated, carbon fibres in a vacuum of $\leqslant 10^{-5}$ Torr at temperatures up to 650° C.

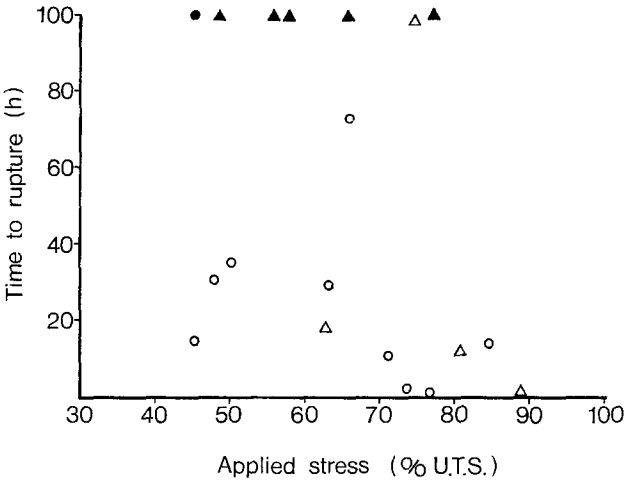

Figure 3 Effect of applied stress on the time to rupture of 180 nm aluminium coated HT-S carbon fibres (▲, 600°C; ○, 625°C; filled symbols represent unbroken fibres).

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